Report for 2002TN3B: Acid Catalyzed Hydrolysis of Wastewater Activated Sludge for Removal and Possible Conversion to Products

- Articles in Refereed Scientific Journals:
  - Perkins L W, K T Klasson, P Bienkowski, R M Counce, 2003, Preliminary Investigation of Nitro-Hydrolysis for Wastewater Sludge Treatment, Separation Science and Technology for Energy Applications, Vol. 38, Nos 12 & 13, pp. 3273 3288.
  - Perkins L W, K T Klasson, R M Counce, P Bienkowski, 2003, Development of Nitrolysis for Excess Sludge Treatment-A Factorial Study, Industrial and Engineering Chemistry Research, 42 3544-3548.
  - Perkins L W, K T Klasson, R M Counce, P R Bienkowski, 2003, Development or Nitrolysis for Excess Sludge Treatment II: A Factorial Study for Industrial Wastes, Industrial and Engineering Chemistry Research (in press).
  - Perkins L W, K T Klasson, R M Counce, P R Bienkowski, Reaction Rate Development and Analysis for the Nitrolysis of Municipal and Industrial Excess Sludge, Chemical Engineering Communications (submitted for publication).
- Other Publications:
  - PRESENTATIONS -Kentucky-Tennessee Water Environment Association Conference, Marriott Hotel, Knoxville, TN, May 13-17, 2002, Nitrolysis as a Sludge Treatment Alternative -American Nuclear Society Spectrum 2002: 9th Biennial International Spectrum Conference, Reno, NV, August 4-8, 2002, Preliminary Investigation of Nitrolyisis To Treat Biosolids from Wastewater Treatment -2002 AIChE Annual Meeting, Indianapolis, IN, November 3-8, 2002, Preliminary Investigation of Nitro-Hydrolysis for Wastewater Sludge Treatment -University of Tennessee, Chemical Engineering Dept. Seminar, March 25, 2003, Design and Development of a Process for Acid Hydrolysis of Excess Sludge -2003 AIChE Annual Meeting, San Francisco, CA, November 16-21, 2003, Development of Nitrolysis As An Alternative For Treatment Of Excess Sludge Produced During Industrial Wastewater Treatment Activities

Report Follows:

Both municipal and industrial treatment of wastewater using an activated sludge process generates large quantities of biosolids referred to as sludge. Currently the Knoxville Utility Board (KUB) generates 65 tons/day of these solids (dry basis) from their activated sludge wastewater treatment facilities. This material is concentrated from 4.2 wt % up to 35 – 40 wt % via filtration and disposed off-site by trucking it over 70 miles for disposal through land farming. Every year the distance becomes greater due to KUB's inability to find acceptable sites for land farming. At least several industries (DuPont and Tennessee Eastman) have similar problems with sludge disposal. Eastman currently produces 55 dry tons/day of sludge which is incinerated.

On site destruction of the excess biosolids is preferred from both an economic and environmental standpoint. Currently both Dupont and Tennessee Eastman use on site incineration and would prefer a more environmentally benign process which uses less energy. A nitric acid catalyzed hydrolysis process can convert most of the sludge into a biodegradable material suitable for recycle. There is also a possibility that the sludge could be converted into acetic acid for commercial sale. The major products from this hydrolysis are organic acids. It may be possible to optimize production of these acids to the point were it is economically feasible to convert the waste activated sludge stream into a commercial product. If it is not economically feasible to recover the organic acids the stream can be recycled back to the waste treatment unit where the organic acids will biodegrade.

This proposal will consist of a batch scale kinetic study using activated sludge from KUB's Kuwahee treatment facility, employing a factorial experimental design. The variables consist of residence time, reaction temperature, solids concentration, and nitric acid concentration; with percent conversion and acetic acid concentration as the dependent variables. The concentration of the sludge can have a significant impact on the economics of any potential process and must be investigated. The sludge stream from KUB's activated sludge treatment process is 4.2 wt % and is concentrated to 35 – 40 % by filtration prior to land farming. A feed sludge concentration in the range of 4.2 - 40 wt % will be investigated. The data will be analyzed and a mathematical model will be developed for the reaction kinetics over the range of the input variables. The form of the model will depend on the experimental data. If significant quantities of organic acids are obtained a complex model will likely be required; if the organic compounds are not produced in sufficient quantity a much simpler model describing destruction kinetics will be employed. The model will be utilized to evaluate the economics of the process and to develop a preliminary process design. A continuous pilot scale unit will be designed and built. This unit will be used at KUB's Kuwahee treatment facility in the second year of the project.

The First Year Objectives for this research are:

1. A complete data set on the reaction kinetics and development of a mathematical model from this data set for design and optimization
2. Development of a process flowsheet for evaluation of economics
3. Economic potential for recovery of products
4. Complete construction of a 2.0 gph pilot plant capable of continuous operation at the optimum batch reactor conditions.

(7) Methodology and Accomplishments to Date:

During the first year of this project a factorial experimental design was developed for conducting batch kinetic experiments on activated sludge effluent obtained from KUB. The objective of the experiments was two fold; i) to hydrolyze the biosolids to CO_2 and organic material which is biodegradable via recycle to the waste treatment unit and ii) investigate the possibility of producing organic acids that maybe recovered commercially. Four variables were used in the experimental design; i) reaction temperature, ii) residence time, iii) acid concentration and iv) biosolids concentration in the feed. The feed solids range was already been fixed by the sludge availability at KUB (4.2 – 40 wt %). The other three variables are all interrelated. The idea of a factorial experimental design is to set up a four dimensional matrix covering the anticipated range of each of the four control variables, but to only run selected experiments. It would be prohibitive to run all of the possible experiments, instead some initial experiments will be conducted and based on these results (percent reduction of biosoilds and/or quantity of organic acids produced) the direction for future experiments will be determined. This direction can be determined either by numerical regression of the experimental data or by fitting the data to a predetermined kinetic model. Obviously what we want to find is that combination of these four variables that leads to high percentage destruction of biosoilds and possibly high conversion to organic acids. After several experiments were conducted it was determined that it was more useful to combine acid concentration and sludge concentration into one variable, acid/sludge ratio.

(8) <u>Principal Findings and Significance:</u>

The volume and viscosity of both municipal and industrial sludge can be significantly reduced with an acid based hydrolysis at 160 to 180 C with a residence time of 5 to 10 minutes. The effluent stream is suitable for recycle to the wastewater treatment facility. Kinetics have been developed for this process based on batch reactor data. Production of an acetic acid product requires considerably longer residence times and results in a product concentration so dilute as to make recovery uneconomical. Several process flow sheets have been developed to convert the bench scale experiments into a viable process. A continuous pilot plant with a capacity of 2.0 gph of a 4 wt% sludge has been designed and constructed for the purpose of demonstration. The results of the first year of this project are documented in the publications and presentations listed below.

(9) <u>Future Research and Funding:</u>

Eastman Chemical Company and The University of Tennessee have received a $982,500.00 NICE3 grant from DOE (DE–FG44-03R410870) for a commercial demonstration of this technology at the Tennessee Eastman plant site in Kingsport. The grant is for 3 years, 6/23/03 – 6/22/06 with $480,000 from DOE and the remained in matching from Eastman and UTK.